CLAIMS

WHAT IS CLAIMED IS:

1. A process for preparing haloalkanes which comprises:

a) mixing and heating carbon tetrachloride with an olefin in a reactor in the presence of metallic iron, dissolved iron species, and an organophosphate co-catalyst to produce a continuous flow of reactor effluent containing a haloalkane product,

b) treating the reactor effluent from step a) with a solid-liquid separation device that separates coarse solid iron particles from the liquid and returns the course solid iron particles to the reactor, to produce by this separation a reactor effluent containing a decreased concentration of coarse solid iron particles,

c) distilling the reactor effluent from step b) in a catalyst recovery unit to produce a continuous flow of overhead product containing an increased concentration of product haloalkane and a continuous flow of bottom product containing increased concentrations of dissolved iron and phosphorus-containing species that are active in the catalysis of the reaction to make product haloalkane, and

d) recycling a portion of the bottom product from step c) to the reactor while purging the rest from the system.

2. The process of claim **1** wherein the organophosphate co-catalyst is trialkylphosphate.

3. The process of claim **2** wherein the trialkylphosphate co-catalyst is tributylphosphate or tripropylphosphate or triisobutylphosphate.

4. The process of claim **1** wherein the main inputs to the reactor are: carbon tetrachloride, ethene, tributylphosphate, metallic iron, and recycle catalyst from the catalyst recovery unit, and the product haloalkane is 1,1,1,3-tetrachloropropane.

5. The process of claim **1** wherein the solid-liquid separation device is a centrifuge, a hydrocyclone, a filter or a sedimentation tube.

6. A process for preparing haloalkane which comprises:

a) mixing and heating carbon tetrachloride with an olefin in a reactor in the presence of metallic iron, dissolved iron species, and an organophosphate co-catalyst to produce a continuous flow of reactor effluent containing product haloalkane, and

b) treating the reactor effluent from step a) with a solid-liquid separation device that separates coarse solid iron particles from the liquid and returns them to the reactor, to produce a reactor effluent containing a decreased concentration of coarse particles, and

c) distilling the reactor effluent from step b) in a catalyst recovery unit to produce a continuous flow of overhead product containing an increased concentration of product haloalkane and a continuous flow of bottom product

containing increased concentrations of dissolved iron and phosphorus-containing species which are active in the catalysis of the reaction to make product haloalkane, and

d) distilling the bottom product from step c) in a continuous or batch secondary catalyst recovery unit to produce an overhead product containing an increased concentration of product haloalkane and a bottom product containing a increased concentrations of dissolved iron and phosphorus-containing components that are active in the catalysis of the reaction to make product haloalkane, and

e) recycling a portion of the bottom product from step d) to the reactor while purging the rest from the system.

7. The process of claim **6** wherein the organophosphate co-catalyst is trialkylphosphate.

8. The process of claim **7** wherein the trialkylphosphate co-catalyst is tributylphosphate or tripropylphosphate or triisobutylphosphate.

9. The processes of claim **6** wherein the main inputs to the reactor are: carbon tetrachloride, ethene, tributylphosphate, metallic iron, and recycle catalyst from the catalyst recovery unit, and the product haloalkane is 1,1,1,3-tetrachloropropane.

10. The process of claim **6** wherein the solid-liquid separation device is a centrifuge, a hydrocyclone, a filter, and a sedimentation tube.

11. A method of producing a haloalkane comprising reacting ethene, carbon tetrachloride, tributylphosphate, and iron in a reactor to produce a reaction effluent, separating the reaction effluent by distillation into an overhead fraction of desired haloalkane, ethene and carbon tetrachloride and a bottom fraction of catalyst components for return to the reactor, in the distillation step of the effluent, the overhead containing 1,1,1,3-tetrachloropropane as well as ethene and carbon tetrachloride.

12. In the process of claim **11** wherein the ethene and carbon tetrachloride are separated by distillation and returned to the reactor.

13. In the process of claim **11** wherein the bottoms fraction, which contains 1,1,1,3-tetrachloropropane, 1,1,1,5-tetrachloropentane and higher molecular weight compounds, is subjected to distillation at a temperature of 70 to 115° C and at a pressure of 40 to 225 torr to obtain the purified 1,1,1,3-tetrachloropropane.

14. The process of claim **13** wherein the distillation temperature is 80 to 105° C and the pressure is 62 to 160 torr.

15. The process of claim **14** wherein the distillation temperature is 93° C and the pressure is 103 torr.

16. The process of claim **13** wherein the bottoms fraction is returned to the reactor after removal of the 1,1,1,3-tetrachloropropane.

17. The process of claim **11** wherein the bottom fraction is subjected to a second distillation at a temperature of 70 - 115° C after the first distillation at 70 – 115° C in order to produce an increased concentration of 1,1,1,3-tetrachloropropane and the residue of the distillation containing catalyst components may be recycled to the reactor.

18. The process of claim **11** wherein the reaction in the reactor is carried out at below 150° C.

19. The process of claim **18** wherein the process is carried out at below 130° C.

20. The process of claim **19** wherein the reaction time in the reactor is between 0.2 and 20 hours.

21. In the process of claim **11**, the liquid residence time is 0.2 to 20 hours; the temperature is 90 – 130° C and the pressure is 30 – 200 psig.

22. A method for preparing 1,1,1,3-tetrachloropropane comprising the steps of

a) reacting ethene with carbon tetrachloride, in the presence of a metallic iron and ferric chloride catalyst and a tributylphosphate co-catalyst.

b) separating the solids from the liquid reaction mixture,

c) distilling the liquid reaction mixture at a temperature of from 70 to 115° C, and at pressure of from 40 to 225 torr overhead pressure, to produce an overhead fraction rich in 1,1,1,3-tetrachloropropane and a bottom fraction

containing catalyst components, 1,1,1,3-tetrachloropropane, and unwanted high-boiling byproducts.

d) distilling the bottom fraction a second time at a temperature of 70 to 115° C and at 40 to 225 torr to obtain additional 1,1,1,3-tetrachloropropane.